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Fused-Quartz Fibers

A Survey of Properties, Applications, and Production Methods

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Nancy J. Tighe

Fused-silica fibers have an important function in many measuring instruments used in scientific research. Much of the information on the production and fabrication methods and on the properties of the fibers is widely scattered throughout the technical literature. This Circular is a survey of this literature and a summary of the findings. A bibliography of pertinent references on the subject is included to provide the sources of more complete and detailed information necessary for specific applications.

1. INTRODUCTION

This Circular is the result of a literature survey covering the properties and the uses of fused-silica fibers. Silica fibers or, as they are frequently called, quartz fibers, have had wide application in precision measuring instruments despite the fact that the factors which affect their behavior are not fully known. The survey of the literature revealed that while some investigations are made in order to obtain specific values of particular properties, other studies are made primarily to develop more completely the theoretical treatment of glasses and their relation to liquids and solids. Although this Circular covers primarily those properties related to the actual use of the fibers, it mentions those theories and properties needed for better understanding of the variations observed in the testing and use of silica fibers. It is hoped that this summary will aid in the production of silica fibers having more reproducible and predictable properties.

Throughout the literature such terms as fused silica, fused quartz, vitreous silica, silica glass, quartz glass are used to refer to types of fused silica. The use or misuse of the terms has been discussed frequently [7, 112, 301, 303 to 305][1] but little uniformity has resulted. The terms used are meant to indicate the degree of transparency or the source of the raw material. For instance, in the glass industry the term fused silica may refer to the transparent material formed by the fusion of quartz sand or other forms of silica, while the term fused quartz may refer to the transparent material formed by the fusion of pure quartz rock crystal. However, this designation of terms is not used consistently, for the material is frequently described as clear or translucent fused quartz or as clear or translucent fused silica. In this Circular the term fused silica will be used in general for that glass formed by the fusion of silica. Where necessary, this term is modified by the adjectives clear or translucent. For instance, where the condition of transparency is evident from the use, such as in fibers which are drawn from clear fused silica, the fibers are referred to simply as silica fibers.

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[1] Figures in brackets refer to the references listed in the bibliography.

2. APPLICATIONS OF FUSED-SILICA FIBERS

Although the first fused-silica fibers were made by Gaudin in 1839 [64] and some tubes and spirals were made and exhibited by Gautier in 1869 [67], these articles were more curiosities than useful tools. It was not until about 1887 when Boys [1] drew fibers and used them in his gravimetric work [65] that their usefulness was exploited. Today silica fibers in the form of paper, matting, cr wool are used in sizable quantities in heating and insulating devices [77, 80, 313]. These devices are well suited for high temperature work because of the high-softening range, low-heat conductivity, and resistance to thermal shock exhibited by the fibers. However it is in the usage of single fibers that the unique properties such as high strength, elasticity, thermal, electrical, and chemical resistance are most fully utilized. It is with these individual fibers and their adaptability to precision measuring instruments that this review is chiefly concerned.

Individual fibers, with or without metallic coatings, have been used as suspensions, as sensing elements and as unit assemblies in many precision instruments. Silica fibers have been used as galvanometer suspensions [1, 65], in gravimetric balances [65, 66], in electroscopes and electrometers [9, 70, 74], in low pressure manometers [78], in radiameters [75], in ionization chambers [79], and in magnetometers [73]. They may possibly be of some use as electrodes in electron tubes.

One of the more extensive uses of silica fibers is in the microbalance field. The entire weighing system can be made of fibers, thus avoiding any errors due to differing densities and coefficients of expansion of the members. Silica fibers are particularly desirable for these instruments because of their high strength in tension and in torsion, nearly perfect elasticity, negligible hysteresis. Further, the resistance to chemical attack, low rate of sorption, low permeability, and ease of cleaning allow such balances to be used under many difficult weighing conditions. Only brief mention is made here of the major developments in fiber microbalances. Reviews of silica fiber microbalance developments [12 to 18, 50] and papers on specific balances should be consulted for complete information about theories and principles of operation and design.

The earliest microbalance using silica fibers was that designed by Salvoni [19]. This type of balance [20 to 24] relies on the rigidity of a cantilever fiber to indicate displacement due to load. Shortly afterward Nernst [25] introduced a microbalance consisting of a horizontal silica fiber attached to and supporting a fine glass rod with a counterpoised pointer to indicate deflection due to load. This type of balance, which uses a silica fiber as a knife edge, is still in use although in forms slightly modified from the original [26 to 36]. Steele and Grant [37, 38] were the first to construct an arm balance entirely of fused silica. The balance, made from fine fused silica rods with fiber load suspensions and a fused-silica knife edge, utilizes a method of weighing by pressure. Several modifications in the design or in the operation of the Steele-Grant balance have been made for adaptation to particular uses [39 to 42, 46, 48]. Pettersson [43 to 45] introduced suspension of the arm balance by two fine vertical fibers as a replacement for the conventional knife edge; and developed further the theory of such suspension. Neher [9] designed a microbalance which consisted of a silica-fiber crossarm attached to a horizontal silica torsion fiber, the twist of which is proportional to the load. This balance can be brought to an equilibrium position by rotating the torsion fiber with a wheel calibrated to indicate the amount of twist in mass units.

From this point microbalances using silica fibers were usually designed by combining certain principles developed in the earlier fiber balances. The balance of Kirk and Craig [23, 47, 49] and modifications by Carmichael [53], El-Badry and Wilson [50], and Garner [54, 310] consist of a nominally equal-arm beam with fiber pan suspension and use a torsion fiber to determine weight differences. These balances differ slightly in design and operation, the greatest design difference being in that of Garner which has a single vertical suspension fiber in addition to the torsion fiber.

The simplest and apparently most widely used fiber balance is the helical spring type [55 to 63], frequently referred to as a McBain-Bakr balance [55]. The silica spring balance can be used for sorption measurements, density determinations, measurements of heat loss and evaporation. Discussions of design characteristics [107] and application to particular measurements can be found in the references cited. The springs resist corrosion and can be easily cleaned but have very little damping due to internal friction.

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3. PRODUCTION AND FABRICATION METHODS

3.1 Fused Silica

One of the greatest drawbacks in the commercial use of fused-silica fibers has been the difficulty and expense of preparing the bulk product from which the fibers are drawn. Prior to World War II no fused silica of good optical quality was produced commercially in the United States [87]. However since that time many of the technologic difficulties have been overcome and fused-silica rods are available in many grades and sizes.

The two forms of fused silica, transparent and nontransparent, are products of quartz rock crystal and of quartz sand, respectively. Sand contains occluded gases and impurities which are difficult to remove entirely in the cleaning process or in the fusion process with the result that the fused product contains volatile and nonvolatile impurities of approximately 0.06 and 0.1 percent, respectively [8]. These impurities are revealed in the masses of tiny bubbles which make the product nontransparent in varying degrees. On the other hand quartz rock crystal can be selected with few if any internal impurities and can be more effectively cleaned so that the occluded gases and other impurities on the surface are removed [87]. The resulting product is relatively free of bubbles and is highly transparent. Quartzites, natural deposits of quartz and vein quartz, which contain more impurities than rock crystal, are poor substitutes for rock crystal; they do however make a better product than quartz sand for the nontransparent form of fused silica [8]. A more recent development is the production of fused silica from "noncrystalline" materials [293,314].

The production and forming methods for fused silica in no way resemble those for commercial glasses [87]. This is because of the high temperatures needed for complete fusion of the highly viscous melt, complicated by volatilization. Although the melting point of quartz is probably below 1,470°C, the rate of fusion of quartz and the rate of change of the resulting glass into cristobalite are about the same below about 1,500°C. Above this temperature the formation of glass is able to keep ahead of the crystallization so that as the temperature is raised rapidly to above about 1,700°C there is no tendency to crystallize [7,109]. The fused product at this point is a soft plastic mass becoming fluid only after the temperature is raised to the range 2,000° to 2,500°C rapidly enough to prevent too much loss due to volatilization [109]. The development and refinement as well as the difficulties of fusion techniques for fused silica are described in several review articles [4, 7, 10, 85 to 88, 109] and in the original works of Shenstone [3], Paget [6], and Hutton [82].

Silica can be fused in arc furnaces, resistor furnaces, graphite molds, and by directed flame. Each of the methods has certain difficulties including those caused by losses from volatilization and by reduction in the presence of carbon and hydrogen. With carbon or graphite electrodes or molds, carbon monoxide is formed and silicon carbide and elemental carbon vapor or silicon metal may be formed [88]. A purer product is obtained if the fusion is done in an oxidizing atmosphere which negates the effects of the reducing tendencies of carbon. There is no tendency for quartz or fused silica to adhere to the carbon if both are pure, for the oxides of carbon prevent close contact between the molten silica and the carbon electrodes or molds [82,88]. However, particles from the electrodes, molds or from the walls of the furnace deposited in the melt cause infusible hard zones which impair the quality of the final product. Further refinements of the fusion techniques may eliminate this type of impurity.

3.2 Fused-Silica Fibers

With the developments in the fusion and forming techniques for fused silica, some of the difficulties of fiber production have been overcome. Early workers with fused-silica fibers [1 to 3, 92] were hampered by the lack of commercially available stocks of transparent rods from which fibers could be drawn. By fusing small pieces of fused silica into the form of a rod and working this rod until it was fairly smooth, these men obtained a rod from which fine sticks and then fibers could be drawn. This was a tedious and time-consuming process and limited the quantity and quality of fibers which could be drawn in a reasonable time. Threlfall [2] mentioned spending 14 days to get two fibers about 2.5 microns in diameter by about 13 inches long.

The production of silica fibers may be classified by two major methods. One method includes those processes used for limited production of single fibers. Namely, blowing of fibers with a flame and the drawing of fibers by hand, and by simple mechanical means whereby the length is limited or the amount drawn is small. The other method includes those processes in which automatic or semi-automatic machines are used for continuous production of single fibers.

The process of blowing fibers in a flame depends on the action of friction of the gases used. Two slightly different methods are used, one [9, 195] producing a long fine fiber, the other [2, 92] a mass of short fibers. In the first process the stock, a fiber about 25 microns in diameter, is held vertically in a long flame until a finer fiber is blown out. The size of the fiber thus blown depends on the size of the original stock, the temperature and size of the flame and the time interval between starting the fiber and its removal from the flame. The fibers are smooth in appearance but have a slight curl which decreases the strength [195]. In the second method the fiber stock is first drawn apart in the flame and the two pieces stroked back and forth in the flame, which is placed in a horizontal postion. The fibers are blown out horizontally and caught on a cloth or board placed a few feet from the flame. These fibers are deposited in a tangled mass and must be eased apart. They are fairly short and may be damaged by superficial scratches from the contacts. Fibers can also be drawn by a hand method [1, 5, 9] in which the stock is melted at a point and the two ends drawn rapidly apart to produce a rather heavy fiber; or by the gravity method [94] in which the stock, weighted at one end, is heated at a point until melted sufficiently so that the falling weight draws out a fiber. Boys [1] and Threlfall [2] each developed a simple mechanical method for drawing fibers. Boys used a crossbow with a straw arrow which when released drew from the fiber stock a fine fiber as long as 60 feet. Threlfall used a modified catapult the slider of which when released drew short thick fibers.

Machines for continuous drawing of silica fibers generally have three elements: a chuck to hold the silica stock stationary, to rotate it about its axis, or to move it lengthwise into the flame; a holder to maintain the torch at the proper angle and either to hold it steady or to move it along the fiber stock; a rotating reel to draw the fiber from the molten stock at a given rate [11, 102 to 104, 107]. These elements can be geared to be regulated independently or as a unit in order to draw a desired size of fiber under readily reproduced conditions. A convenient base for the elements is a lathe or a drill press used either horizontally or vertically. This type of machine has been used to draw fibers from 600 to 0.7 microns from stock of 12 to 0.15 millimeters in diameter. Once the flame is adjusted to give sufficient heat for thorough melting of a particular size of stock then the reel speed and the rate of feed of the stock into the flame can be related to the approximate diameter of the fiber [11, 103]. The ratio of fiber pulling to fiber melting can be regulated within limits above which the fiber breaks off and below which the stock is not sufficiently melted for uniform drawing [11]. Drive slippage and speed recovery accompanied by alternate drawing of cold and hot silica into the fiber results in variations in strength and in the diameter of the fiber [103].

Many gas mixtures have been used in all of the above methods: oxy-hydrogen, oxy-coal gas, oxy-natural gas, oxy-acetylene. It has not been mentioned whether the gases used affect the fibers, although the heat of the flame must be regulated so that it is neither high enough to volatilize an excessive amount of the fused silica nor low enough to prevent sufficient melting. With the thicker stock, curly fibers result from uneven heating [1, 2, 94, 195], so curly in some cases as to resemble watch springs [2]. These fibers can be straightened by "soft flaming" but are weakened as a result [195]. A slight bowing due to the effect of gravity on larger fibers has been noticed when they are drawn horizontally; however, the small fibers can be drawn either horizontally or vertically with no noticable effects [316].

With flame-blown silica fibers about 21 microns in diameter and 15 cm in length, Horton [185] observed variations in the diameter of about 1 to 4 percent. The fibers had the appearance of a sequence of irregular conic sections within an over-all tapered fiber. Similar irregularities in shape of about six percent in a quarter of an inch were observed in some machine drawn fibers [316]. The long term variations could be associated with variations in the stock and with the pulling phase. For example, rungs or tapes used on sectional reels cause an irregular rate of pulling which results in a slight taper in the fiber between the markers [103], a variation which is especially noted in fine fibers. The short term variations in the diameter could arise from insufficient melting of the stock, oscillations of the flame, vibrations in the drawing equipment, or even a natural oscillation between the rate of drawing and the rate of melting. Although these diameter variations were not mentioned by many workers, it is possible that they were not sought or that they were so small as to be hardly perceptible with the measuring equipment used.

Although fused silica fibers seem to lose strength with age [188 to 191, 199, 200] the real effects of actual storage conditions on this loss of strength are not known. It would appear that a method of storing fibers might be determined which would help to preserve the strength and to prevent damage by dust, moisture, certain atmospheres, or accidental scratching.

Contamination and scratching of the fibers from dust, salts in the air, and from handling with the fingers tends to weaken the fibers. When the fibers are fused or heated the silicates formed by impurities on the surface can cause brittle joints and weak fibers [50, 315, 316]. Finger marks on large pieces of fused silica appear as devitrification marks on the material after it has been worked in a flame [300]. Opinions regarding methods to counteract this contamination and subsequent weakening differ among workers. Some workers prefer to prevent contamination by working under scrupulously clean conditions, using only those fibers which are freshly drawn, free of dust and other contamination and by never touching that part of the fiber which is used in a piece of apparatus [9, 315]. Others prefer to remove any contamination on the fibers by cleaning with a solution, such as chromic acid, before the joints are fused or the fibers worked [104, 106]. The finished piece is also cleaned to remove any deposits which result from handling during the working of the fibers. The effects of cleaning solutions on the strength of fibers and on the finished piece are not known or have not been detected. The effects are complicated by the fact that while cleaning, rubbing with the fingers, and adsorption of moisture reduce the tensile strength, these processes tend to increase the resistance to scratching or to any further surface injury [113].

3.3 Silica-Fiber Apparatus

The fineness of the silica fibers used necessitates the use of holders or jigs to prevent the fibers from blowing away while they are being fabricated into the various devices. A number of different types of holders and jigs are used. These aids, used mostly in the fabrication of microbalances and electrometers, are described more thoroughly by the individual experimenters.

The holders used by Neher [9] and by Carmichael [315] consist of forks with two prongs whose spacing is either adjustable or is fixed for a particular length of fiber. These forks hold individual fibers only at the ends of the fiber and allow the fiber to be held in any position while joints are fused. The holders can be held in a micromanipulator, or the proper sized holder can be placed in position in a jig designed to insure reproducible construction of a particular device. The used portion of a fiber is never touched so that any damage which may result from contact with a holder or with the fingers is avoided. With this type of holder the fibers can be removed directly from a storage reel, thus eliminating extra handling steps.

Kirk [104,311] used two types of holders; one is a holder with multiple prongs on which a fiber is supported and shaped; the other is a Y-shaped holder on which a fiber is supported. Fibers are clipped or fastened to these holders at intervals along the usuable length and can then be held in any desired position. A predesigned assembly of these holders allows for repeated construction of fiber apparatus. However, where the holders are made of heavy silica fibers or other hard materials there is some danger of scratching resulting from the contact between holder and fiber.

In some applications where fairly heavy fibers are used for a beam or supporting member, metal, carbon, or asbestos blocks appropriately grooved are used as templates. The fibers are laid in the grooves, the joints fused or cemented and the fine fibers either drawn out from the assembly or fused on in the appropriate places [28, 32, 35 to 37, 39, 40]. Joints made in this way are frequently brittle and weak because of overheating and may be contaminated by the block itself [50].

Two methods using templates as patterns or guides in the construction process have been used. The templates used by O'Donnell [102] consist of thin glass plates equipped with pegs to locate the fibers and with silica fiber clips to hold the fibers in place for fusing. The plates are held in proper positions in jigs so that all the joints can be fused through the appropriate holes drilled in the plates. El-Badry and Wilson [50] used a series of patterns drawn on matt paper. Fibers attached to microscope slides are placed in position over the pattern and the joints cemented in successive steps until the assembly is completed. All the fibers can be attached in this manner.

While there are certain disadvantages to the use of some of the methods such as contamination, overheating of the joints, scratching, the ease of manipulation and the character of the final assembly may outweigh any disadvantage and thus determine the method to be followed in individual applications.

In the construction of spiral or helical springs, long even silica fibers are needed. Although shorter fibers have been fused together to make a fiber long enough for a spring [106] this practice may affect the performance of the spring.

Silica springs are wound on a suitable mandrel by feeding the fiber in place and heating it to conform to the shape of the mandrel. The mandrel may be tapered for ease of removal and to suit the design characteristics of the spring [107]. Several materials have been used for mandrels including carbon and graphite [55, 95 to 98, 100, 101, 106], Pyrex [105], Vycor and fused-silica [99, 107] rods or tubes. Of these, fused silica and Vycor mandrels have been considered most suitable because they have the slight adherence necessary to prevent stretching and a coefficient of expansion the same as or similar to that of the fibers wound [107]. Carbon and graphite mandrels may damage the spring by overheating and by contamination. Springs wound on carbon or graphite mandrels may have squared rather than smooth coils.

Over the years many methods of winding silica springs have been developed. In the earliest successful method the mandrel was turned about its tilted axis by hand and, as the fiber coiled about the mandrel, a flame bent it to the mandrel [55, 95, 106]. This method has been greatly improved and mechanized to produce more regular springs [97, 98, 100, 105]. In the latest and most completely mechanized method, as described by Ernsberger and Drew [107], the mandrel is turned in a lathe which has controls for the fiber and flame guides to allow for uniformity in the spacing and in the coils, and for proper heating of the fiber.

As with all procedures for fabricating apparatus from silica fibers, the precautions against contamination and damage resulting from handling and from the actual fabrication process should be observed.

For many applications a conducting surface of metal or of graphite [79] on silica fibers has proved very satisfactory. There are several methods for putting this coating on the fibers including sputtering, evaporating, plating, or baking a painted coat [2, 5, 79, 91, 94]. A coating or plated surface on the ends of fibers has also been used to facilitate holding fibers in certain apparatus.

Additional equipment for fiber work includes a binocular microscope, micromanipulators and holders, torch holders, small torches made from fine fused-silica tubing or from hypodermic needles. Detailed descriptions of the techniques of construction of fiber devices and descriptions of the equipment used in their fabrication are given by Neher [9], O'Donnell [102], Haring [103], and Kirk [104]. Individual construction details and specialized design problems are discussed most completely by the workers who developed the apparatus (see applications).

In regular fusing operations involving quartz or fused silica [299, 300] the intense heat radiation, the fumes and disagreeable odors cause fatigue and nausea among most workers. For these reasons and because of the possibility of silicosis most workers in this country work only 2 or 4 hours with quartz or fused silica. Special glasses such as those using Noviweld lenses should be used to protect the eyes from the intense infrared radiations. A suitable exhaust system should be used to carry away the heat and the fumes.

While these discomforts have been referred to in large scale operations there is little reference to such effects when working with silica fibers. This could be because of the smaller masses involved and the consequent smaller amounts of heat and fumes. It is well to note, however, that there are some precautions to be taken in quartz fusing operations.

4. MECHANICAL PROPERTIES OF FUSED-SILICA FIBERS

4.1 Strength

Strength is defined as the resistance of a material to fracture or quantitatively as the critical value of stress at which fracture occurs [223]. The mechanical strength of fused silica can be considered in three categories: compressive strength, bending strength, and tensile strength. The compressive strength of fused silica is 2 or 3 times that of ordinary glasses and is about one fifth less than that of quartz [7]. The bending strength, modulus of rupture, of silica fibers is generally higher than the tensile strength [7, 188 to 190, 241] and is affected by the same factors which cause so much scatter in the values of the tensile strength. The tensile strength of silica fibers is smaller than the crushing strength. However, the individual values vary much more than the uncertainty of measurement which makes comparison of values difficult.

The tensile strength of silica fibers exceeds that of most metals and other materials. This high strength coupled with an almost perfect elasticity makes silica fibers desirable for many applications. However the great range of individual values for strength and the slight loss of strength due to certain conditions of use prevent or limit those applications needing more than the minimum values of strength. For this reason there is a need to investigate the strength of silica fibers with special attention given to the methods of production and to the conditions under which they are used. Most measurements of tensile and bending strength on any quantity of silica fibers [188 to 195] were made with fibers produced by flame, hand, bow and arrow or other simple methods. The only available measurements on machine drawn fibers [11] as published are not very complete.

The scattering in the determinations of tensile and bending strength of fibers is so large that the assignment of a definite strength is difficult. A graph showing experimental values obtained for tensile strength and for bending strength might appear to be of some help. However an analysis shows dissimilarities in individual testing procedures, in the specimens, handling, thermal treatment, temperature and humidity controls, in the test equipment and in the presentation of the results; all of which tend to affect the values determined for strength and thus make any strict comparison misleading. The results of the various investigators show trends in the strength such as an increase in strength with decreasing diameter and length, some variations due to the atmosphere, to thermal treatment, to age, to drawing methods, to handling methods. The possible errors that may result from taking values at random from experimental curves can be illustrated by a plot of all the previously determined values of tensile strength with respect to the diameter, on a log-log scale. Here where a value of the strength for a 100-micron fiber is about 35 to 40 kg/mm², the tensile strength given for a 10-micron fiber may range from 2 to 12 times larger than this value, while those for a 3-micron fiber may range from 5 to 100 times larger than the values given for the 100-micron fiber. The trend on these curves for fibers over 100 microns is a slow approach to a constant value, while that for fibers less than 100 microns is a rapid increase in strength in a linear manner.

In the experimental curves showing the strength of silica fibers as determined by an investigator different methods of showing the results are used. Such curves may indicate maximum values [188 to 191], values adjusted for the presence of flaws [195], average values [192, 193], values without the conditions specified [11], and in some cases all the values determined [1, 2, 188 to 190]. Without some standardization of the testing procedure, comparisons of such values are frequently meaningless. For example the diameter measurements can greatly affect the results [307, 309] especially with small diameter fibers. An interesting discussion of the problem of correlation of strength measurements on glass is presented by Bailey [207].

The mechanical strength of glasses has been termed a "structure sensitive" property [213]. The random structure with the resulting variations in bond strength and in the interatomic distances gives glasses and especially fused silica an initially high value for strength. However, the theoretical strength is somewhat modified by certain discontinuities in the structure and by the effects of various external factors on them [116, 213]. The differences between the theoretical and the experimental strength of glasses led Griffith [199, 200] to postulate the existence of certain concentrations of energy in the form of submicroscopic cracks or flaws throughout the material. These flaws could occur either during the manufacturing process or in subsequent treatment of a glass. The effect of flaws or discontinuities is to produce local stresses which exceed the average stress and as a result lead to failure. Calculations of the effects show that they could conceivably account for the differences between theoretical and actual strength [200, 208, 223]. Proof of the existence of flaws was demonstrated in experiments [163, 164] which showed fine cracks in places on annealed fused silica rods and tubes where no accidental scratching could occur. The Griffith flaws appeared to be about 10 microns long by about 0.01 microns deep.

a. Production

The drawing process for silica fibers has some effect on the final strength. In regard to the method of production fibers drawn by bow and arrow appear to have higher tensile strength and show less scattering of values than those blown in a flame which in turn appear to have higher tensile strength and show less scatter than those fibers drawn by hand [195]. Comparative tests with machine drawn silica fibers are not available in the current literature. It is entirely probable that such fibers could show greater strength and less scatter because of the controlled conditions under which they can be drawn.

The drawing process can be regulated with respect to drawing temperature, rate of drawing, and rate of cooling and thus determine to some extent the strength [103]. The severe quench from the rapid rate of cooling provides a fire polished surface which resists chemical reactions and decreases or delays the effects of cracks or flaws. Recently the importance of the rate of cooling on commercial glass fibers was brought out by Slater [227] who found little variation in strength between fibers 0.002 in. in diameter and those 0.0002 in. in diameter when both were drawn at the same rate of cooling. The rate of cooling can be regulated in the fiber drawing machines by adjusting the drawing temperature and the drawing speed within certain limits [11]. The thermal history of fibers as determined by drawing conditions is one of the important single factors in the strength of silica fibers.

From further considerations of the drawing process two theories were developed in order to explain the difference in strength between fibers and the bulk product from which they are drawn. One theory was advanced by Murgatroyd [172] who suggested that in order to allow the continuity of the fiber as drawn the strongest bonds were selected from the melt and alined parallel to the direction of drawing while the weakest bonds were alined perpendicular to the direction of drawing. In the other theory Bickerman and Passmore [220] believed that the flaws in a fiber must be oriented favorably to enable the fiber to be drawn. These theories emphasize the importance of the drawing process rather than the reduction of size which accompanies it. Although there are many who support or have supported these ideas [143, 149, 173, 213, 215, 222], recent experiments [148, 161] on annealed glass fibers indicate that there is neither an orientation of bonds nor one of flaws in fibers which could be responsible for the high strength. Additional evidence showed that an oriented structure was not evidenced by X-ray studies [160] and that if an orientation at the surface existed, its influence on the strength could not be detected [193].

b. Size

Experiments on the breaking strength of silica fibers show an increase in strength with decreasing fiber diameter [1, 11, 188 to 195]. Similar results occur with other glass fibers [195, 235] and in glass slabs where a small area is tested [236]. An additional increase in strength is found when the specimen length is decreased [190,220]. Assuming that flaws exist in fibers, then the apparent dependence of strength on fiber size can be explained in a statistical manner by the decreasing probability of effective flaws occurring as the volume or the surface of a fiber is decreased [193, 213, 214, 220]. Although there is some basis for assuming a relation between the probability of flaws occurring in relation to the size of a specimen, an exact expression for the distribution function of the cracks or flaws is difficult to formulate [223].

Orowan [223] considered that the necessarily arbitrary assumptions as to the size of cracks and to the number of cracks in a given volume or over a given area without accounting for the individual history of the cracks led to results "more interesting mathematically than correct physically." However, the thinnest silica fibers which have been investigated have diameters less than the supposed size of an ordinary Griffith crack and the strength of these fibers appears to approach the theoretical values determined for the material. These fibers could not contain flaws as large as those in larger specimens and unless the flaws were large enough to weaken the fiber by reducing its cross section, the strength, Orowan concluded, should be higher than that of a thick rod. The consideration of the number and size of flaws in a given length particularly affects measurements of bending strength where the probability of flaws occurring at a certain point of maximum stress is smaller than that of flaws occurring over a given length, with the result that values for bending strength are generally higher than those for tensile strength. In addition, the nonuniformity in strength over the entire length of a fiber [241] due to the existence of flaws is a possible cause for the great range of values determined for any one size of fiber. A well-designed statistical test procedure might help to remedy the situation.

c. Surface

The large surface area in comparison with the volume of fibers may account for the noticeable effect of the surface and its treatment on the strength of silica fibers. The chemical and physical nature of the surface of glass has been described as different from that of the body of a glass [160]. Structurally, this influence of the surface can be explained by a broadening of the bond-strength distribution near the surface which tapers off toward the interior and leaves weak bonds at the surface. Although flaws are considered to be distributed throughout the material, the strength is determined by the most dangerous flaws on the surface [193, 199, 200], for a surface flaw can exert twice the stress of the same sized inner flaw [221]. The stress variations in individual fibers resulting from the existence of flaws, scratches on the surface, and the effects of surrounding conditions on them, may cause fracture before the theoretical elastic limit is reached. Because of the effects on strength of these conditions it is difficult to bring values of individual fibers within a small range of error. It may be appropriate to mention here the often quoted idea of Little that one measures not the strength of a material but the weakness of its surface. A discussion of the chemical and physical aspects of the surface condition of glasses is presented in a series of works by Weyl [116, 175, 213].

d. Environment

Glass fibers are tested and used under adverse conditions because of the character of the material. For the actual normal conditions of atmosphere are corrosive to commercial glasses and to some extent to fused silica just as certain special atmospheres are corrosive to metals and other materials [213]. Experiments on strength of fused silica fibers in certain controlled atmospheres indicate a relationship between moisture absorbed and the strength [192, 193].

After silica fibers have been exposed to the air, water vapor is absorbed which decreases the strength from that obtained immediately after the fiber is drawn. When this layer of moisture is removed the tensile strength increases. The tensile strength of fibers, which have been heated in a vacuum to remove all absorbed moisture, increases up to 3 or 4 times the strength in water or water vapor. When the same or similar fibers were dried in $CaCl_2$ the strength increased about one and a half to two times the strength in water vapor [190, 192, 193]. When water or alcohol vapor was admitted into the evacuated apparatus the strength immediately decreased to its original values. For fibers of a given diameter the strength in oil, and in alcohol is greater than that in air. In addition, the strength of those fibers which were in the air for a long time was the same as that of fibers broken in water, and the strength of fibers stored in a room saturated with oil or alcohol vapor was the same as that of fibers broken in the liquids.

Further experiments [188 to 193] on the effects of etching in hydrofluoric acid revealed a 3 to 5 fold increase in strength of silica fibers after the etching. There was, however, a considerable amount of scattering of the values which was partially explained [193] as a result of the etching process which alternately smoothes and then exposes flaws; the strength thus depending on the condition of the surface at the time of removal from the acid. The strength of fibers broken in the acid did not differ much from that of fibers broken in the air. Furthermore, the strength gradually increased with etching until a certain maximum thickness had been removed, after which no further increase was noticeable [190]. These etched fibers were very susceptible to damage from the air or from scratching although a protective coating of shellac helped them retain their high strength [193].

The strength of silica fibers is decreased by scratches from atmosphere dust [199, 200] and from other fibers. There is an immediate loss of strength resulting in fracture when a fiber under strain is touched by another silica fiber. A further decrease of strength results from contact with the fingers [213, 315], although those parts rubbed with the fingers are protected from further injury [113]. It has been suggested occasionally that silica fibers be coated to prevent damage from the various atmospheric conditions or to increase the strength. There is at present no published data available on the effects of coatings on silica fibers. However, investigations [195, 227] of the effects of coatings on textile glass fibers revealed that while the coating itself did not increase the strength it did lessen the effects of atmospheric attack in some instances. The chief use of a resin or wax coating is to prevent seizing of glass fibers when they are woven or pressed. Silica fibers are much more resistant to damage than commercial glass fibers and in present applications careful handling will avoid much harmful damage. A protective coating may prove useful however.

The tensile strength of silica fibers increases slightly with increasing temperature up to the flow region, showing minimum strength at room temperature [108, 116]. This can be explained by the fact that the healing of the cracks by surface diffusion as the temperature is raised outweighs the influence of thermal motion which tends to increase tension and to spread the cracks [213]. In addition, the absorbed moisture layer which is thick and active at room temperature is driven off at higher temperatures and is inactive at low temperatures [213]. Experiments [10] showed an increase in strength of silica fibers after they were heated to 1,188°C for 4 hours and then allowed to cool. However, heating silica fibers in a flame makes them weaken considerably [2]. Although the strength may change with temperature changes, the strength at relatively low temperatures does not depend on the temperature [193].

The values of strength thus vary depending on the conditions of test and of use. Any improvement in these conditions should increase the apparent strength. A strength test gives not the true value of strength but rather the residual value after the fiber has been damaged in some way [114].

4.2 Elastic Properties

Silica fibers exhibit almost perfect elasticity up to the breaking point; any deviations are so slight as to be barely measurable.

Although the elastic constants have been determined since silica fibers were first used [1], there is not complete agreement between early values and those determined rather recently. Again as with strength, testing methods and conditions of the test and of the specimens considerably affect the final results with the errors either magnified or diminished. Absolute determinations of elastic constants require accurate values of size, density, and mass [244]. Temperature effects must also be considered if individual measurements made under different conditions are to be related or compared. Anelastic effects which cause the strain to lag behind the stress, must be accounted for since the elastic constants are related harmoniously only if the rate of stress application is the same [224].

Within the experimental error, the elastic moduli can be considered to remain fairly constant over the range of sizes used. The experiments of some investigators [1, 2, 5, 9, 188 to 191] show a dependence on the fiber diameter of Young's modulus and of the shear modulus. However, later investigations showed that these moduli were independent of the diameter and of the length, and pointed to slight errors in former measurements [11, 193].

A summary and discussion of much of the experimental work on the elastic constants of fused-silica fibers can be found in Sosman's treatise on silica [7]. Some of the experiments [1, 2, 182, 185], and later work [184, 187, 188 to 191, 244] show that Young's modulus and the shear modulus do not vary appreciably over the range of temperatures used in the tests. There is a slight linear increase in both of these constants with increasing temperature up to the region of viscous flow. The moduli continue to decrease from values measured at room temperature to those measured down to -200°C [245]. The change in shear modulus with change in temperature affects the readings of very sensitive measuring instruments [62, 63, 107]. For example, Sheft and Fried [62] found an increase in rigidity, in a spring balance, of about 0.02 percent per degree centigrade which was reflected in a change in elongation of 0.02 percent of the total load per degree centigrade. The change seemed to indicate strengthening with increasing temperature.

The elastic moduli vary with the surrounding atmosphere, exhibiting higher values in a vacuum and in alcohol vapor than in water or moist air [192], which increase could not be due to the weight of the moisture. This effect was also observed in silica springs where an increase in elongation occurred in water or alcohol vapor but not in inorganic vapors when these springs were exposed to the vapors after being in a vacuum [196]. This was, however, erroneously attributed to an expansion of the silica because of absorption. Silica fibers etched in hydrofluoric acid had values of elastic moduli in close agreement with unetched fibers with some uncertainty regarding an actual increase [192].

Slight deviations from "perfect elasticity" of silica fibers can be attributed to a slight delayed elastic effect noted when fibers are twisted through large angles. The classification of the types of deformation due to applied load generally used are: an instantaneous strain which is completely recoverable, a delayed elastic strain which is recovered slowly, and a viscous flow which is not recoverable but which appears to be nonexistent in fused silica at temperatures below about 800°C [7, 117]. The delayed elastic effect is similar in effect to a progressive increase in viscosity and frequently is confused with a viscous flow or creep [117]. Although delayed elastic effects are of some concern in commercial glass fibers, the effects in fused-silica fibers are rather negligible, approximately 100 times less than in other glass fibers [217, 218]. A delayed elastic effect has been noticed in instruments using a torsion fiber. However, a constant correction could be applied, as after a short time the rate became constant. In addition the effect decreased with decreasing fiber diameter so that in the finest fibers the effect is barely perceptible [1, 66]. A part of the apparent delayed elastic effect could also be attributed to the mountings used for the fibers [24, 53]. With increasing temperature the delayed elastic effect, expressed as the ratio of the delayed strain to the instantaneous strain, increases. Fused silica has the lowest value of the ratio of all the glasses [117].

5. SOME PROPERTIES OF FUSED SILICA

The properties of fused silica observed at room temperature combine to make it such a useful material. While some properties may change with time, temperature, or surrounding atmosphere the magnitude of the changes is usually small compared with similar changes in other materials. The properties discussed in this section usually are determined for fused silica in bulk form, but are of some importance in the use of silica fibers.

Since fused silica is a glass, the most simple glass, it would seem appropriate to place glass in reference to other states of matter. The ASTM describes glass [301] simply as "an inorganic product of fusion which has cooled to a rigid condition without crystallizing." The vitreous or glassy condition is rather difficult to define as is evidenced by the numerous papers on the subject some of which attempt to describe the condition of glass as distinct from or similar to the solid and liquid states [124 to 164].

5.1 Structure

The present picture of the atomic arrangement of fused silica developed from the laws of crystal chemistry, analysis of X-ray diffraction patterns, and studies of certain physical properties. It was from studies on crystal structure that Goldschmidt [126], Zachariasen [126], Sosman [7], and others [124, 151, 155] developed theories on the atomic arrangement and formation of glasses. The early methods and the apparent similarity between crystal and glass led some observers [124, 151] to believe that fused silica and other glasses were made up of particles of crystalline material called "crystallites." However, even without the analytical tools later used by Warren and his co-workers [127 to 134], Zachariasen postulated the existence of a random three-dimensional network with energy comparable to that of the corresponding crystalline network in fused silica and glasses in general and laid down certain conditions as necessary for the formation of oxide glasses. The network picture thus postulated by Zachariasen was later verified by Warren and his co-workers. These experimenters using a Fourier analysis of the X-ray diffraction pattern, first of fused silica and later of other glasses, were able to show not only the arrangement but also the average interionic distances and the mean bond angles. They also showed that any particles in fused silica showing similarity to crystals are too small to be described as crystalline matter.

The structural arrangement of fused silica as confirmed by Warren's analysis consists of a short order structure which is tetrahedral in form with 1 silicon ion bonded to 4 oxygen ions, with each oxygen ion bonded in turn to 2 silicon ions. The tetrahedra share the oxygen corners to form a long range three-dimensional random network. A two-dimensional picture of this structure shows the network made up of a series of irregular rings, where the average number [154] of tetrahedra per ring is six and the number of tetrahedra in individual rings varies from 3 to 10 or more. Where the bond angle between tetrahedra is nearly 180° and varies slightly for successive tetrahedra, a buildup of a random or "disordered" [143] network is allowed. It is this flexibility in bond angles which gives fused silica and other glasses the long-range disorder of a liquid; while the ordered distances and angles within a tetrahedron gives them the short-range order of a crystal.

5.2 Chemical Durability

At room temperature, fused silica is attacked by hydrofluoric acid. Near 300 to 400°C, phosphoric acid starts to attack silica. At high temperatures and up to 1,000°C, weak and moderately concentrated solutions of basic salts and of metallic oxides and basic salts react with fused silica. Fused silica is reduced to silicon by carbon at temperatures over 1,600°C [110]. It is also reduced at high temperatures by hydrogen which forms silicon hydride. When this material is cooled the part of the silicon hydride not oxidized decomposes into silicon and hydrogen and forms a deposit of silica and silicon on the surface of the material [281]. This deposit, observed on fibers fused in a flame containing hydrogen, has frequently been confused with devitrification of the fiber.

5.3 Permeability

Fused silica is permeable to neon, hydrogen, and helium at elevated temperatures and under pressure [7, 10, 109, 112, 296]. Devitrified and nontransparent types of fused silica are more permeable than the transparent form. The rate of diffusion may depend to some extent on the size of the openings in the random network [116, 296] and on the size of the gas atoms. Thus fused silica with larger random openings than other glasses would allow larger gas molecules to pass through and the smaller gas molecules could pass through more quickly than in some other glasses [296]. Reviews of work on permeability indicate considerable differences in observations on the amounts of diffusion through fused silica [4, 109, 112].

5.4 Sorption

Fused silica exhibits a combination of a reversible adsorption plus a slow permanent sorption of water [285]. This reversible adsorption was reduced about 35 percent by acid treatment, and an additional 30 percent by heat treatment. The rate of permanent sorption as found by Barrett [285] in finely powdered transparent fused silica, fell off rapidly in about a half hour after exposure to moisture and then became constant at about 4×10^{-10} g/cm²/hr. This process seemed to be an actual diffusion of water into the silica. Fused silica apparently does not adsorb inorganic vapors in sufficient amounts to be measurable [196]. It does however adsorb alcohol vapor [192, 193] and the vapor of paraffin oil [175] though in smaller amounts than the water vapor. This adsorption of moisture affects the strength and the elastic constants such as Young's modulus and the shear modulus which in turn affect the behavior of measuring instruments [62, 106, 196].

5.5 Hardness

The "hardness" of fused silica is not only a function of the strength and the elastic properties but must be greatly influenced by the chemical resistivity of the glass and by the environment [116]. The hardness of fused silica has been determined on the various hardness scales although an exact definition has not been formulated. The Mohs' hardness numbers given for fused silica range from 5 to 7 [9, 10, 312]. The Knoop indentation hardness number given for fused silica is approximately 475 [284] where the value for quartz is approximately 710 to 790 [302]. This Knoop indentation scale shows fused silica to be harder than some other glasses and many metals including brass, gold, silver, tantalum, and some stainless steels.

5.6 Density

The density of transparent fused silica is approximately 2.21 g/cm^3 while that of nontransparent fused silica is about 2.07 g/cm^3. The density of the specimens of fused silica may vary about 0.05 percent or even as much as 0.1 percent, a variation of about 10 times that of specimens of crystalline quartz [7]. Sosman thought that it might be possible to correlate these variations with the source, with the thermal treatment or with the state of strain in the fused silica. Since that time these factors have been related to density changes in a number of experiments [170, 289, 292].

Density measurements at temperatures up to 1,700°C showed that there is an equilibrium density which increases with temperature [289]. The specimens used changed from the initial density to the equilibrium value for the temperature at which the specimen was heated. The rate of change increased as the temperature increased so that at 1,300°C they had a density corresponding to the high-temperature density. These changes in density with heat treatment were found to occur in addition to the normal thermal expansion, in effect a slow change of volume in addition to an instantaneous change in volume.

Under uniaxial pressure an increase in density was noted [289, 292]. Bridgman and Simon [292] found a threshold pressure near 100 atm above which the structure of fused silica seemed to collapse. Rapid increases in density of 7.5 percent and occasionally at high as 17.5 percent, to 2.61 g/cm^3, were found. After X-ray examination showed the specimens to be amorphous, the increase was attributed to a folding up of the network; a bending of the Si-O bonds rather than a shortening of them. This folded-up structure was mechanically stable at ordinary temperatures, but with heat treatment the density decreased to the original value. Both Douglas [289] and Bridgman and Simon concluded that there appears to be an equilibrium configuration which may be approached from either direction. Their experiments support Sosman's theory that heat treatment and strain could cause variations in individual specimens.

5.7 Devitrification

The rate of crystallization or the devitrification of fused silica depends on the temperature to which it is heated, the period of heating and on the degree of subdivision. It is also affected by atmospheric dust [251], by the previous thermal treatment [7], and may be related to the viscosity [248].

While fused silica is thermodynamically unstable at all temperatures below 1,710°C, its molecular sluggishness (viscosity) at room temperature is so great that no change toward crystallization has been observed at such temperatures [7]. Fused silica devitrifies only after prolonged heating at high temperatures. At 1,000°C [7] or even at 1,200°C [10] devitrification is hardly perceptible. Above these temperatures fused silica slowly devitrifies into cristobalite. When samples were powdered, almost complete devitrification occurred after heating at 1,100°C for 6 days or at 1,600°C for 1 hour [83].

Although devitrification could not be called a purely surface phenomenon [112], it does start at a surface and work inward [7, 108]. Specimens with a great deal of surface area such as powdered specimens, or those with a large number of bubbles such as translucent types devitrify more readily than large pieces and transparent fused silica [7, 108, 183, 249].

A possible form of devitrification occurred when fused-silica articles were exposed to radium salts at the temperature of boiling water but did not occur at lower temperatures [246].

A dependence of the rate or of the amount of devitrification on previous thermal treatment is evidenced by the appearance of less devitrification on specimens made from high temperature melts than on those made from lower temperature melts [7], as well as a greater tendency toward devitrification exhibited by annealed specimens.

The presence of fluxes such as calcium carbonate, calcium oxide, liquid silicates, and certain metallic oxides hastens devitrification [253]. Although titanium oxide and zirconium oxide have been added to fused silica to decrease any tendency toward devitrification, the resulting glasses devitrify more rapidly than pure fused silica [7].

5.8 Thermal Expansion

Investigators have long been interested in the thermal expansion of fused silica, which is smaller than that of almost all other materials, because of its usefulness in measuring instruments. Critical examinations of the experimental work on expansion of fused silica presented by Sosman [7] and by Souder and Hidnert [260] show that differences in testing equipment and in methods of testing are responsible for many variations observed in the results of many investigators. With transparent and nontransparent fused silica tested over a range of -125° to 1,000°C, Souder and Hidnert determined a critical temperature of about -80°C where specimens had a minimum length. Viscous flow which started around 800°C hindered their measurements of expansion above that temperature.

The expansion of fused silica changes slightly with heat treatment. Heat treatment over the range 20° to 750°C increased the expansion by about 20 percent of the total expansion [170] which results in a slightly larger coefficient for annealed specimens than for unannealed ones [260]. In addition to the change in expansion, a permanent increase in length occurs after heat treatment. An average variation after cooling from 1,000°C was 0.001 percent with a maximum of 0.003 percent [108, 260].

The coefficient of expansion of transparent fused silica is slightly larger than that of the nontransparent [108, 255, 260], a difference amounting to about 25 ppm on the heating cycle and to about 30 ppm on the cooling cycle [108].

Thermal expansion or the instantaneous change of volume with change of temperature [289] can be explained by structural considerations and defined in terms of thermal history and the temperature of measurement. The small coefficient for fused silica as compared to that of quartz could be a direct result of the random structure of the glass [133, 166]. Since a small thermal expansion can take place by a change in average interatomic distance or by a configuration change such as a change in bond angles, an increase of disorder or an increase in structural binding would decrease the expansion coefficient [289], where bond angle changes in the random structure can compensate for other effects which tend to increase the coefficient [153]. The negative coefficient is due to an increase of structural binding with decrease in temperature [166, 170].

5.9 Viscosity

It is principally with reference to the development and improvement of methods of melting and working glasses that the relationship between viscosity and temperature, time, composition, and heat treatment have been studied. Much of the work done on viscosity is with specimens of commercial glass types, for studies on fused silica are limited by insolubility and by the high temperatures required to decrease the viscosity enough for present experimental conditions [306].

At room temperature and below several hundred degrees centigrade the viscosity is so high that the material is considered as solid; for instance, the viscosity at room temperature has been estimated at about 10^{60} or 10^{70} poises which is rather incomprehensible for practical meaning [267]. Preston thus considered that viscosity has an upper limit of definition as well as a lower limit, with a viscosity over 10^{14} approaching infinity as a quantitative measure. Even in the melt the viscosity is so high that fused silica never really becomes fluid [10]. The viscosity of fused silica at room temperature is so high that movements toward an equilibrium configuration (stabilization) or a configuration appropriate to the temperature at which the glass is used are prevented [117]. The high temperature configuration of the rapidly cooled fibers is retained permanently for all general purposes. The fibers have what is referred to as a low viscosity compared with slowly cooled fused silica, a difference which may account for the differences in properties between the two forms [117].

Studies of the viscosity of fused silica and other glasses are incomplete. The process of viscosity changes due to thermal treatment and the effects of this on the properties of glasses are not completely known. A survey and discussion of the available work and its implications was made by Jones [117]. This and other articles [174, 267, 268, 270, 276 to 280] should be consulted for details of the developments and problems in studies of viscosity.

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